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A Strain State in Synthetic Diamond Crystals by the Data of Electron Backscatter Diffraction Method

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Abstract—The authors put forward a procedure of determination of strain tensor components through the analysis of distribution of intensity of back-scattered electrons in Kikuchi patterns. The strain state has been studied in local areas of a synthetic diamond crystal produced by the temperature gradient method in the Fe–Al–C system through growing onto a diamond single crystal synthesized in the Ni–Mn–C system. Characteristic surfaces of the strain tensors and strain ellipsoids have been plotted; special features of strain distribution in the crystal have been analyzed. Diagonal tensor components have been determined from the changes of distributions of intensity of individual bands, the other components have been found from the displacements of axes of zones relative to their positions in the standard Kikuchi pattern.

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1. INTRODUCTION

The Mg–C system is used in diamond synthesis for this provides the possibility of producing diamond crystal of a high degree of crystalline perfection at fairly low process parameters (temperature and pressure) [1]; however, the crystal synthesized in this system are unstable. The process pressure and temperature can be lowered (down to 7.7 GPa and 1700°C, respectively) by adding Ni to the Mg–C solid solution [2]. At the same time, the elements of the Mg–Ni system cause changes to electrical and optical properties of the crystals depending on the amount of these elements [3]. This has been used in practice, especially in electronics, optical filters and devices for measuring absorbed dose rate [4].

The electron backscatter diffraction—the Kikuchi method—is an efficient tool for the determination of a local strain state in monocrystalline materials as well as in polycrystalline films and multilayered structures [5]. The method offers a spatial resolution and localization property owing to the use of advanced CCD detectors [5].

The methods based on the determination of distribution of intensity of Kikuchi bands [6] and displacement of zone axes [7] give information on a change in interplanar spacings along certain crystallographic directions. This permits finding individual strain tensor components. For this purpose, researchers usually apply the methods of cross–correlation of images [8]. In this work we propose a new alternative method for the determination of all strain tensor components, which is based on finding the displacement of axes of zones (intersections of Kikuchi bands) and on an analysis of changes in the distribution of intensity of individual bands.

2. EXPERIMENTAL FINDINGS

The study was carried out on synthetic diamond crystals produced by the temperature gradient method in the Fe–Al–C system at high static pressure (about 6 GPa) and high temperature (about 1700 K), the growth duration was approximately 48 h. For a substrate we used diamond of the Ni–Mn–C system (surface (100)). The Kikuchi patterns were taken from various regions of the sample by means of a Mod. EVO-50 (Carl Zeiss, Germany) scanning electron microscope using a CCD detector. The angle of incidence of an electron beam of diameter ca. 40 nm onto the crystal surface was 70°.

The position of each Kikuchi band was determined using the standard software; in that way, we obtained information on crystallographic orientation of the zone axes (a point where the zone axis emerges at the image plane, which corresponds to the intersection of the Kikuchi bands) [9]. In addition to the standard software we used also the program developed by ourselves, which permits

– plotting profiles of distribution of intensity in the sections of Kikuchi bands [10, 11];

– comparing the coordinates of zone axes in Kikuchi patterns from each region with the coordinates of those from the reference region, and finding their displacement [10, 12];

– calculating interplanar distances along various crystallographic directions for each region [7, 10, 11];

– determining local components of the strain tensor [9, 11].

3. CALCULATION OF STRAIN TENSOR COMPONENTS

It is well-known that a transition from the *XYZ* coordinate system for a strain-free lattice cell to the *X'Y'Z'* coordinate system for a strained lattice cell is given by the relationship [13]

$$\begin{bmatrix} X' \\ Y' \\ Z' \end{bmatrix} = \left(\begin{bmatrix} e_{xx} & e_{xy} & e_{xz} \\ e_{yx} & e_{yy} & e_{yz} \\ e_{zx} & e_{zy} & e_{zz} \end{bmatrix} + \begin{bmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 0 \end{bmatrix} \right) \begin{bmatrix} X \\ Y \\ Z \end{bmatrix} \tag{1}$$

or, if in a matrix form, by

$$A' = (M + I)A,$$

where M is the matrix of the displacement gradient, which can be split into a symmetrical and antisymmetrical parts (the strain tensor and the rotation tensor, respectively), I is the unit matrix.

Due to a strain the local region in a crystal, which is given by a vector **r**, will be shifted into a new position **r′** by a vector **Q**. In this case, on the screen of the CCD detector the translation vector **Q** is mapped into a vector **q** (Fig. 1). The last-mentioned vector is a quantitative characteristic which is determined from Kikuchi patterns, i.e., represents the displacement of zone axes relative to the position they would take in a Kikuchi pattern from the reference region of the sample, or the distances between two zone axes. This is what is used for finding the strain tensor in local regions of a sample.

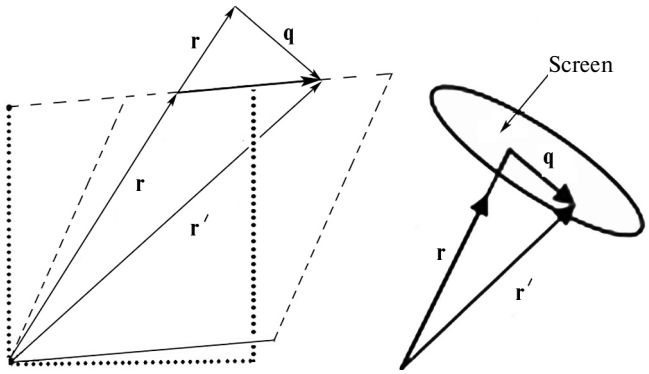


Fig. 1. A schematic representation of a displacement of an arbitrary vector **r** in the crystal and a projection of the displacement on the Kikuchi pattern.

It follows from Fig. 1 that

$$\mathbf{q} = \mathbf{Q} - \lambda\mathbf{r}, \tag{2}$$

where λ is the projection constant that depends on the experimental conditions. Upon simple mathematical manipulations, equation (1) in view of (2) takes the form

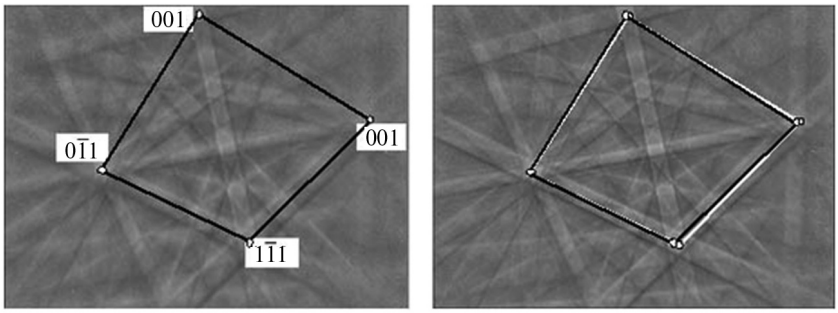
$$\begin{aligned} q_x &= xe_{xx} + ye_{xy} + ze_{xz} - \lambda x; \\ q_y &= xe_{yx} + ye_{yy} + ze_{yz} - \lambda y; \\ q_z &= xe_{zx} + ye_{zy} + ze_{zz} - \lambda z, \end{aligned} \tag{3}$$

where *x, y, z* are the coordinates of the vector **r**; e_{ij} is the respective components of the strain tensor.

The total strain tensor contains rotation and strain components and can be split into symmetrical and antisymmetrical parts. The symmetrical one describes normal strain components. The antisymmetrical part is the rotation tensor and is responsible for the crystal rotation. With the rotation tensor being zero, the system of equations (3) is simplified (due to withdrawal of the projection constant λ) to the form [8]

$$zq_x - yq_z = [yz(\varepsilon_{xx} - \varepsilon_{zz}) + xz\varepsilon_{yx} + z^2\varepsilon_{yz} - xy\varepsilon_{zx} - y^2\varepsilon_{xy}];$$
$$zq_y - xq_z = [xy(\varepsilon_{xx} - \varepsilon_{zz}) + yx\varepsilon_{xy} + z^2\varepsilon_{xz} - x^2\varepsilon_{zx} - xz\varepsilon_{zy}]. \quad (4)$$

The system (4) contains eight equations and nine unknowns, i.e., one of the strain tensor components is an uncertain quantity. To solve the system (4) we need four individual measurements of the displacements q_x and q_y (Fig. 2). In the standard cross–correlation methods only two displacements of zone axes are determined from a Kikuchi pattern for a selected local region of a sample [8]. Thus, for the determination of all strain tensor components the standard approaches need an analysis of a great number of Kikuchi patterns taken from local neighbor regions. Our proposed rapid procedure requires no analysis of patterns taken from neighbor regions and is based on a study of displacements of four axes of the zone in the same Kikuchi pattern, which are also projections of q_x and q_y. For a single-valued solution of the problem various approaches can be applied. One of them implies that a diagonal tensor component, e.g., ε_{xx}, can be found from the analysis of changes in distribution of intensity of individual bands in a Kikuchi pattern. In this case, all the strain tensor components are calculated in the standard crystallographic system of coordinates—[100], [010], [001].



(a) (b)

Fig. 2. Kikuchi patterns taken from two neighbor regions in the crystal (white circles represent the points where the crystal zone axes emerge at the image plane; the length of the segments that connect the points is given by the respective interplanar distances forming a closed contour): reference region (a) and region under study (b); Fig. 2b shows the divergence between the contours taken from these regions (the white contour corresponds to the reference region).

The accurate positioning of zone axes is an intricate and multiple-value problem for a fragment of the image in the vicinity of an intersection of Kikuchi bands (see Fig. 2) has no pronounced peak and there is no symmetry in the distribution of intensity near the intersection. Therefore, we applied the procedure of determination of coordinates of the point where each of the zone axes emerge in the Kikuchi pattern, which was put forward and tested in [10]. Generally, the most perfect region in a crystal is taken as the reference one [6]. Finding the degree of correlation between the fragments of the pattern, which correspond to the emergence of zone axes for the reference region and the region under study, provides information on local strains in a crystal as well as eliminates any local distortions (that may arise due to experimental conditions) of the regions where the zone axes emerge. To implement the correlation method for processing experimental images we developed a Delphi-based software [10] that digitally processes the Kikuchi patterns taken from the reference region and the region under study. Coordinates of accurate positions of the zone axes are found under the condition of minimizing the correlation kernel, accurate to one hundredth of a pixel. This accuracy is ensured just by the correlation element of the method [6]. Using the above-mentioned software we analyzed the band intensity distribution and the positions of zone axes in the Kikuchi bands for 16 local regions in a diamond crystal at hand. The analysis has demonstrated that the Kikuch pattern taken from the fifth local region shows the best fit to the most perfect region of the crystal and can be chosen as the reference one for our case (Fig. 2). This has enabled us to determine the displacements of the respective intersections for other local regions relative to the reference one and to find (by numerically solving the system of equations (4)) five components (all except ε_{xx}) of the symmetrical strain tensor in each of the regions. On the other hand, the diagonal component ε_{xx} is determined from the analysis of the change in the intensity profile for individual bands in the Kikuchi pattern [7]. Thus, we have all the six (nine) components of the strain tensor for each local region in the crystal.

4. CHARACTERISTIC SURFACES AND ELLIPSOIDS OF STRAINS

For geometrical interpretation of principal directions and values of the symmetrical second-rank tensor, in particular the tensor of strains ε_{ij}, we use the notion of the characteristic Cauchy surface which is a second-order algebraic hypersurface centered at the origin of the Cartesian coordinate system [12]:

$$f(x,x) = \tau^{ij} x_i x_j = \tau^i_j x^i x_j = \tau_{ij} x^i x^j = \text{const}. \quad (5)$$

If the ε_{ij} strain tensor is referred to the principal axes, i.e., brought to the diagonal form, then Eq. (5) is written as

$$\gamma_x (x)^2 + \gamma_y (y)^2 + \gamma_z (z)^2 = \text{const}, \quad (6)$$

where γ_x, γ_y, γ_z are the values of the components of the diagonalized strain tensor; const is an arbitrary constant responsible for scaling.

For plotting the characteristic surfaces (Fig. 3) of the strain tensor for each local region of the crystal (Fig. 4) we used the standard software Maple17 (Mapplesoft).

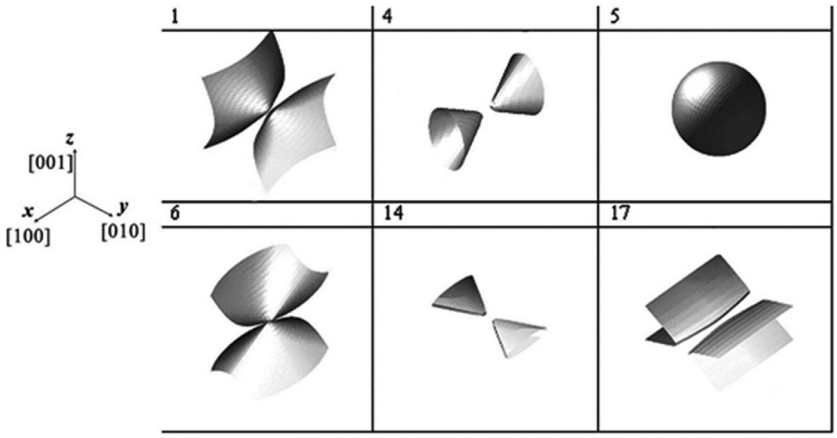


Fig. 3. Characteristic surfaces of the ε_{ij} strain tensor for local regions of the synthetic diamond sample (for regions 2, 3, 8, 12, 13, and 15 the characteristic surfaces are similar to that for region 1 and differ only in the strain value; the characteristic surface for region 9 is the same as for region 4, and that for region 7 is the same as for region 6).

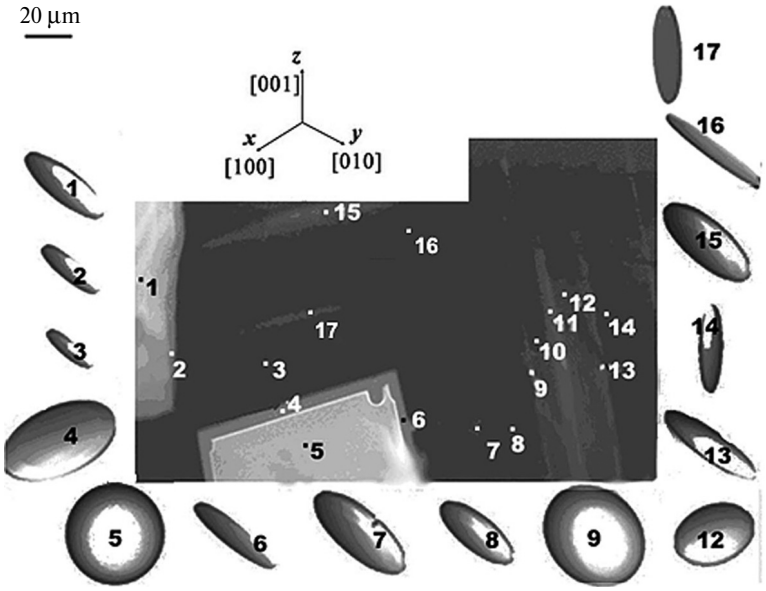


Fig. 4. A cathodoluminescence topogram of the synthetic diamond crystal under study; indicated are the positions of the local regions from which the Kikuchi patterns were taken and for which the respective characteristic surfaces of strain ellipsoids were plotted.

Plotting the characteristic surface of the strain ellipsoid in a unit sphere presentation provides information on the orientational properties of the strain components. This enables us to visually compare the strains active in a certain area. We applied the following relationship to plot the characteristic surfaces:

$$\frac{x'^2}{(1+\varepsilon_x)^2}+\frac{y'^2}{(1+\varepsilon_y)^2}+\frac{z'^2}{(1+\varepsilon_z)^2}=1, \quad (7)$$

where x', y', z' are the coordinates that represent the body's position upon deformation; ε_z, ε_z, ε_z are the principal components of the strain tensor (within the reduced axes $\varepsilon_{xx}=\varepsilon_x$, $\varepsilon_{yy}=\varepsilon_y$, $\varepsilon_{zz}=\varepsilon_z$).

The obtained characteristic surfaces of strain ellipsoids in a cathodoluminescence topogram (Fig. 4) visualizes anisotropy of values of the principal strain components in the sample local regions under study.

The most strained regions in the sample at hand are the following: region 17, with the component $\varepsilon_{zz}=-19\times10^{-3}$; region 11, with $\varepsilon_{xy}=-17\times10^{-3}$; and region 16, with $\varepsilon_{yy}=25\times10^{-3}$. In terms of the component ε_{xx}, regions 1 through 7 are positive while regions 8 through 11 are negative, suggesting a change in the strain field mode between regions 7 and 8. Region 16 located between regions 15 and 17 is the most compressed one along the direction [010], which is indicative of significant strains and an influence of neighbor regions that have strain tensor components different in magnitude and opposite in sign. Also, the component ε_{zz} along the axes [001] in regions 1 through 14 exhibits a periodic change of sign. All the shear components ε_{yz} and ε_{xz} in the regions studied have turned out to be almost equal, positive, and small in magnitude, ca. 10^{-5}, suggesting the absence of any noticeable shear strains (rotations) along the respective crystallographic directions.

5. CONCLUSIONS

We put forward a procedure whereby all the strain tensor components are determined from an analysis of distributions of intensity of reflected electrons in a single Kikuchi pattern. To determine five components of the symmetrical strain tensor we have analyzed the values of displacements of zone axes relative to their positions in the reference Kikuchi pattern taken from the most perfect region of the crystal. The diagonal component (e.g., ε_{xx}) was found through the analysis of the change in the distribution of intensity for individual bands in the Kikuchi pattern.

We have studied the strain state in local regions of a synthetic diamond crystal produced by the temperature gradient method in the Fe–Al–C system through growing onto a diamond single crystal synthesized in the Ni–Mn–C system. Using a comprehensive analysis of the Kikuchi pattern (finding the positions of zone axes, analyzing the Kikuchi band intensity profiles) we have determined the values of strain components and plotted the characteristic surfaces of ellipsoids of strain in local regions of the synthetic diamond crystal. It has been demonstrated that all the regions studied have almost the same values of shear components ε_{xz} and ε_{yz}, suggesting no rotations along the given crystallographic directions, while other components exhibited significant variations. The most strained regions along the directions [001] and [010] are region 17 ($\varepsilon_{zz}=-19\times10^{-3}$) and 16 ($\varepsilon_{yy}=25\times10^{-3}$), respectively. With a reference sample available, the proposed approach will permit improving the accuracy of determination of strain tensor components.

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